PRESIDENT'S ADDRESS

Changing Times and The AOAC*

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The Association of Official Agricultural Chemists has conducted its seventy-fifth meeting; obviously the Association has survived its growing pains. Its work has increased since 1912 when the U.S. Department of Agriculture published Bureau of Chemistry Bulletin 107, which carried the impressive title of "Provisional and Official Methods of Analysis of the Association of Official Agricultural Chemists." Henry Lepper, our Secretary Emeritus, has told us that because of its completeness it "caused disappointment to the young chemists of that day since, through it, all analytical procedures had been perfected for all important foods and determinations." Nothing could have been further from the truth, as we know; the work of the Association has grown to such an extent that the 9th edition of *Official Methods of Analysis* required some 800 pages to cover the multitude of methods that are now official, and serious thought is being given to printing the next edition in two volumes. This rapid growth is the result of the dedicated efforts of the more than 300 chemists and analysts designated as Referees and Associate Referees.

We have seen an increase, not only in the work of the Association, but also in its reputation and the use of its standard methods throughout the years. As Dr. William Horwitz, the editor, has stated in the preface of the 9th edition: ". . . some state laws require the use of AOAC methods, where applicable; . . . federal specifications and private contracts utilize AOAC methods, and AOAC methods have been quite generally accorded a preferred status in court testimony." Equally important is the recognition that AOAC methods of analysis are applicable in research, especially in the fields dealing with composition and utilization of agricultural commodities.

When I was introduced to the AOAC and its "Book of Methods" more than 30 years ago, my first reaction was that of many generations of young chemists before and after that time—a feeling of respect for its members and the utmost confidence in its official methods, but an impression that it was an organization of old men who were lagging far behind and who were putting their stamp of approval only on methods of long standing. I took it for granted that the methods in the Book of Methods were irreproachable; yet to get my job done I had to look elsewhere for the newer methods specific to my problem. Eventually I learned that the latter situation was not in the purview of the Association. The Association was not designed for the introduction of new methods; its constitution clearly states that methods of analysis, to be approved as official, must be subjected to collaborative study, and two years is specified as the minimum time in which this can be done.

The Flexibility of the AOAC

Another lesson the analyst must learn is that even though a method had been written into *Official Methods of Analysis*, the Association's responsibility for that method is not ended. Instead, the Association must constantly be alert to note whether or not a method continues to meet the required standards of reliability, reproducibility, and specificity.

The Association has kept pace with the great advances that have been made in the field of analytical methods, both chemical and physical. *Official Methods of Analysis* contains such new techniques as ion exchange, paper and column chromatography, and absorption-spectrographic methods which include ultraviolet, visible, and infrared. We

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already have a committee serving jointly with the American Oil Chemists' Society on gas phase chromatography and papers on the use of the mass spectrograph and the polarograph have been presented at our meetings. These and other instruments, together with such sophisticated techniques as differential refractometry, sedimentation, and dextran filtration, will soon be included in *Official Methods of Analysis*. Methods for the detection of radioactive contaminants of foods are being discussed this year.

Revision of Methods

In the Association's program of keeping alert and flexible to changing demands, the development of official methods for new drugs, insecticides, food additives, and so on, has been accompanied by revision of older methods. For example, one of the oldest AOAC methods, namely, the Kjeldahl method for determination of nitrogen, has been almost continuously under investigation. This was one of the first methods made official by the Association. In 1943, A. C. Chibnall, writing in the *Journal of Biochemistry,* called attention to his discovery that unless the digestion time, especially in the micro-Kjeldahl procedure, was increased to 8 hours or more the nitrogen values for proteins were low. This came at a time when it was the vogue to shorten the method either by use of new catalysts or by micro methods, and it caused much concern, particularly among research workers. The AOAC recognized that something was wrong and the newly appointed referees on microchemical methods attacked the problem. They re-examined the Gunning-Arnold-Kjeldahl method and developed a modification that appeared to be satisfactory. In their collaborative study in this relatively new area they took some liberties and introduced several new modes of approach. Instead of requesting the collaborators to test only the referees' method, each collaborator was also requested to use his own method, this being the one with which he was familiar. Further, instead of using a natural product as the test material for which the method is generally used, compounds of high purity and representing refractory ring structures, as well as those with simple amino groups, were tried. Use of these pure compounds not only eliminated the possible interaction of extraction and nitrogen determination data but also gave a truer evaluation of the accuracy of the method. Another and more important innovation was the application of statistics to the analysis of the collaborators' data. When coupled with information in the collaborators' questionnaire, it was possible to identify those elements of the various modifications of the Kjeldahl method which were critical as well as those elements which were unimportant. The results of this study were quite remarkable. The statistical treatment of the data made it relatively easy to composite a method that became official in a remarkably short time. The method was automatically streamlined and simplified, and yielded results having an unusually high degree of reproducibility and accuracy. It also disclosed that some of the previous modifications of the method, even when used by our best analysts, produced low results with errors as large as 95% when the sample contained refractory compounds. This plan of collaborative study has been successfully employed to develop and refine many micro and semi-micro methods of organic analysis so that they are now official.

The Specificity of Methods

The need for and the use of methods of high specificity, which have been proved by collaborative study to yield accurate and reproducible results by governmental enforcement agencies, is axiomatic. Unfortunately, too many research chemists in the field of agricultural research, and particularly those who are not trained analytical chemists, do not have sufficient background to judge the different methods they use. Yet they must evaluate the results of their work and make future plans on the analytical data obtained by these methods, even though they may be questionable. This has often proved costly. Therefore, all methods of analyses and especially those used in agricultural research should be proved, and there is no better way than through collaborative study which takes care of inter- and intralaboratory differences. A thorough collaborative study will, in the end, shorten the research effort by sharing the research work that is involved in developing reliable methods, and will produce data which can be treated with the desired confidence. When an agricultural research chemist develops a new method for some particular need, it is highly desirable that it be tested collaboratively, and the AOAC stands ready to help in such a study.

A most unfortunate situation can, and sometimes does, occur when a method which has been tested collaboratively is found not to have the specificity first assumed or implied for the method. The AOAC must constantly re-examine its methods to prevent this situation. Even then it sometimes happens. Let us take, for example, the AOAC chromotropic acid method for the determination of formaldehyde. Unfortunately, this method gives positive tests with other aldehydes, such as the carbonyl alkaline decomposition products of simple sugars. When these are known to be absent, as is usually the case, they obviously will not interfere, and we can say that in its usual application this method is specific. However, the unsuspecting researcher, not being warned of this situation, may use the method for measuring formaldehyde in solutions containing these alkaline decomposition products of reducing sugars and in doing so come up with some provocative interpretations of the data. For example, in a study made of the use of paraformaldehyde to maintain sterility of maple tree tap holes, one group of investigators logically turned to *Official Methods of Analysis*. A method was needed for the determination of residual formaldehyde in sirup produced from sap obtained from paraformaldehyde-protected tap holes. They selected the chromotropic acid method, unaware of the facts that the carbonyl decomposition products of the sugars would interfere and that these carbonyls were present in the sugar solutions to be analyzed. Their results inaccurately indicated a high residue of formaldehyde in the sirup. Then, by applying the test to sirup produced from the control, or non-paraformaldehyde-protected tap hole on the same tree, they again found a high formaldehyde content. This led to the logical though faulty conclusion that formaldehyde or paraformaldehyde was being translocated in the tree, and to still another deduction, that the pellet of paraformaldehyde could be inserted in only one of the holes of a multiple-tapped tree to give protection to all of the other tap holes. This information, if passed on to the maple industry, could have been very costly; the data erroneously indicating high residual formaldehyde in the sirup would have been viewed askance by Food and Drug officials.

The Use of Statistics

Statistics can be employed as an aid in the development or refinement of a method. A more general use of statistics by the AOAC will be the unbiased evaluation of collaborators' data. No longer should it be necessary to make decisions upon visual inspection of the data which can lead and has led to wrong conclusions. Some of our referees already employ statistics to evaluate their data. The Committee on Improvement of the Association has expressed itself by recommending an expanded use of statistics. Organizations such as the American Society for Testing Materials and the American Oil Chemists' Society are adopting a similar program. We now have a committee on statistics which is working to develop sound and practical applications; they will attempt to provide a scheme for evaluating data and, later, a scheme for conducting collaborative studies according to statistical designs.

The AOAC collaborative plan of study has not been changed, for there is no substitute for it. The situation is the same now as it was when the founders of this Association realized that this was the best procedure for testing a method. However, it can be streamlined so that fewer collaborators will be required. The need for and the cost of obtaining reliable methods through collaborative study must be justified. How else could such an organization as the ASTM, which is supported by private capital, continue to exist? In all groups where methods of tested reliability must be used, whether in quality control, regulation, or research and development, the cost of obtaining these methods is shared by utilizing the cooperative aspects of collaboration. This places a relatively small burden on any one person or laboratory. Seldom would an individual organization or chemist find it possible to produce the quantity or quality of data obtainable through collaboration, and under such a wide variety of conditions essential for testing the reproducibility of a method.

Other aspects of collaboration which are seldom discussed are the comradeship and *esprit de corps* that come from working together on a common problem. Typical of this is the fine cooperative spirit that now exists among the chemists of the tobacco industry, which in no small part is due to their serving together as collaborators on methods of analysis for tobacco.

Conclusions

This has been a brief summary of the work of the AOAC. The Association has not delayed nor shirked its responsibility to keep abreast of the ever-increasing and changing demands for the improvement of established methods and the development of new methods. These are prerequisites for the analysis of new foods, food products, food additives, insecticides, pesticides, drugs, and cosmetics as well as for compositional analyses of agricultural commodities and their derived products. As research and development continue in these various fields, the task imposed upon this Association will become ever greater. With the great strides that are being made in automation and instrumentation, the Association must be prepared to utilize these fully. I do not want to appear as a prophet, but I believe that the workload of the Association and the need for the application of greater skills will continue to increase.